IDENTIFICATION OF THERMOPHYSICAL PROPERTIES OF A COMPOSITE ONE-DIMENSIONAL REGION

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Abstract. *This work presents recent advancements on the mathematical model and on the experimental apparatus used for the identification of thermophysical properties of solid materials, in the Laboratory of Heat Transmission and Technology, COPPE/UFRJ. The apparatus consists of two cylinders made of different materials, but with the same diameter, butt-joined to form a single composite cylinder. The lateral surfaces of the two cylinders are insulated, while the bottom surface of one of the cylinders is uniformly heated and the top surface of the other cylinder is insulated. The sensitivity coefficients with respect to the different unknown properties are examined in order to identify small magnitudes and linear dependence among the parameters. The D-optimum approach is used for the design of the experiment in order to estimate properties with low uncertainty. The Levenberg-Marquardt method is applied for the identification of the unknown parameters. Simulated and actual temperature and heat flux measurements are used for the estimation of the thermophysical properties of Teflon. The estimated parameters are compared with the properties obtained with other techniques, including the Flash method.*

Keywords: *inverse problem, parameter estimation, design of experiment, thermophysical properties*

1. Introduction

The identification of thermophysical properties plays an important role in today's research and development in engineering, which deeply relies on computational simulation of physical phenomena for design purposes. Several experimental techniques have been developed in the past for the estimation of thermophysical properties by using steady state or transient experiments. Some of these techniques became ASTM standard methods, like the Flash Method for the identification of thermal diffusivity (ASTM, 2001). However, with the advancement of new engineering materials, faster, more accurate and especially dedicated techniques are required. For the development of such new techniques, the use of inverse analysis of parameter or function estimations, in conjunction with the optimum design of experiments, plays a fundamental role (Beck and Arnold, 1977, Beck *et al*., 1985, Alifanov, 1994, Özişik and Orlande, 2000, Woodbury, 2002, Kaipio and Somersalo, 2004). In fact, this represents a new research paradigm, the applications of which can be found in many recent publications, e.g., (Taktak *et al*., 1993, Dowding *et al*., 1996, Guimarães *et al*., 1997, Oliveira *et al*., 1999, Silva *et al*., 2000, Salas *et al*., 2002, Dantas *et al*., 2003, Mejias *et al*., 2003, Borges *et al*., 2005a, 2005b).

The objective of this paper is to present the advancements over some of our previous works, dealing with the identification of thermophysical properties of solids (Oliveira *et al.*, 1999, Silva *et al.*, 2000, Salas *et al.*, 2002). Salas *et al.* (2002) reported correlated temperature residuals in the estimation procedure, resulting from a non-suitable mathematical model that considered one single region and a constant imposed heat flux. In fact, the electrical resistance utilized by Salas *et al.* (2002) was extremely thick (2.3*mm*) and was not capable of providing a constant heat flux, as assumed in the mathematical model, especially for small times. The advancements over the works aforementioned include: (i) use of a two-region mathematical formulation, that involves the heated solid and the one used for homogeneization of the temperature at the non-heated boundary; (ii) change of the electrical resistance that heats the bodies (the new resistance is 0.2 mm thick); (iii) use of a transducer to measure the imposed heat flux, which permits the utilization of the actual measured heat flux in the properties identification procedure. The mathematical model and the experimental apparatus, as well as the parameter estimation procedure utilized in this work are described next, as applied to the identification of the thermal conductivity and of the volumetric heat capacity of Teflon.

2. Experimental Apparatus

The experimental apparatus used in this work consists of two cylinders, made of Teflon and Aluminum, which are butt-joined, as illustrated in Fig. 1. Thermal grease is carefully spread at the interface between the two cylinders, so that the thermal contact resistance is negligible. The diameter of each cylinder is 100 *mm* and the thickness of the Teflon and of the Aluminum cylinders are 4.4 *mm* and 27 *mm*, respectively. The lateral surface of the composite cylinder is thermally insulated with Styrofoam, with inner diameter of 100 *mm* and outer diameter of 180 *mm*. The open surface of the Teflon cylinder is heated with an electrical resistance, while the open surface of the aluminum cylinder is insulated with a Plexiglas plate of 10 *mm* of thickness. A heat flux transducer is placed between the electrical resistance and the Teflon cylinder. The electrical resistance (133.1 Ω) is 0.2 *mm* thick, while the heat flux transducer is 0.5 *mm* thick, both with diameters of 100 *mm*. Such a diameter was chosen in such a way that, even if lateral heat losses are significant, the temperature at the center of the heated surface deviates by less than 4% from the one-dimensional case. For such a design, a strict case was taken into consideration, involving heat losses by convection through the lateral surfaces to the surrounding air with a heat transfer coefficient of 5 $W/m^2\,^oC$. Anyhow, the heater, the transducer and the cylinders have their lateral surface thermally insulated with Styrofoam (Oliveira *et al.*, 1999, Orlande *et al.*, 2000). The electrical resistance and the heat flux transducer were manufactured by Prof. Saulo Guhts, from the Laboratory of Porous Media and Thermophysics Properties (LMPT), of Federal University of Santa Catarina.

The electrical resistance was connected to a DC power source made by Instrutherm, model FA 3050. In addition to the heat flux measurements, the temperatures at the heated surface, at the interface between the Teflon and the aluminum cylinders and at the non-heated surface were measured with type K thermocouples, 30 gauge. Prior to the experiments, the thermocouples were calibrated in the Thermal Metrology Unit Prof. Roberto de Souza, LTTC/COPPE/UFRJ, by using a dry-bath made by Techne, model TECAL 650 S. The thermocouples and the heat flux transducer were connected to a data-acquisition system made by Agilent, model 34970 A. The readings were taken with a frequency of 60 *Hz*. A picture of the assembled experimental apparatus is presented in Fig. 2.

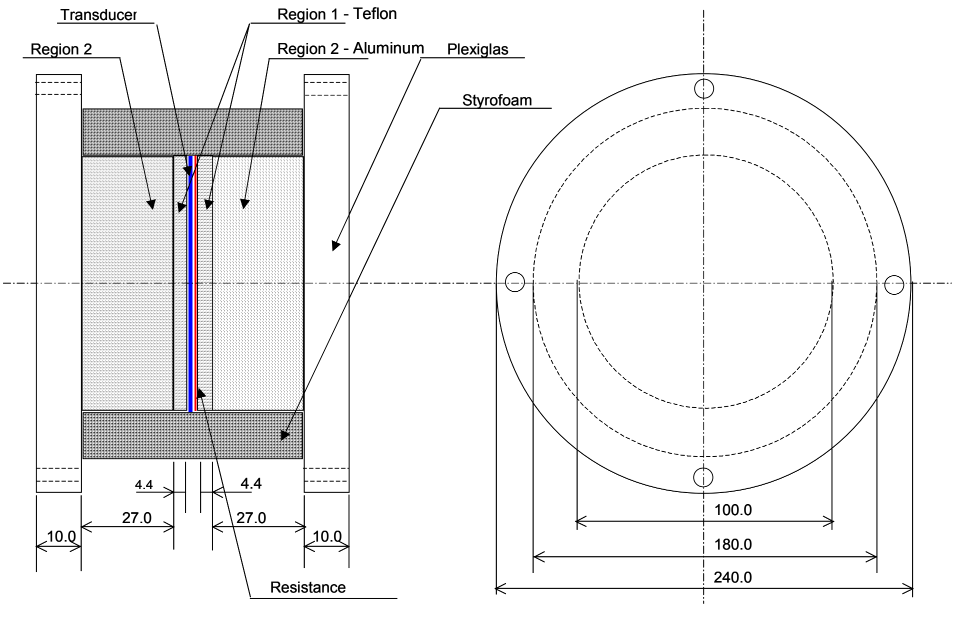

Figure 1. Geometry of the experimental setup

Figure 2. Experimental setup

3. Mathematical Formulation and Direct Problem

In order to describe the above physical problem, we assume that heat transfer through the cylinders is one dimensional and that the contact between them is perfect. For the sake of generality, the two cylinders are denoted as regions 1 and 2, as illustrated in Fig. 1. For the experiment described above, region 1 refers to the Teflon and region 2 to the aluminum cylinders. The thickness of regions 1 and 2 are L_1 and L_2, respectively. The two regions are assumed to be initially at the uniform temperature T_i and, for $t > 0$, the boundary at $x = 0$ is heated with a heat flux $q(t)$ during the heating period $0 < t \le t_h$. The other boundary at $x=L_1+L_2$ is supposed to be insulated. By assuming that the thermophysical properties are constant during the duration of the experiment, the formulation of such physical problem is given by:

Region 1:

$$C_1 \frac{\partial T_1}{\partial t} = k_1 \frac{\partial^2 T_1}{\partial x^2} \qquad \text{in } 0 < x < L_1 \text{, for } t > 0 \tag{1.a}$$

$$-k_1 \frac{\partial T_1}{\partial x} = q(t) \qquad \text{at } x = 0 \text{, for } t > 0 \tag{1.b}$$

$$T_1 = T_2 \qquad \text{at } x = L_1 \text{, for } t > 0 \tag{1.c}$$

$$T_1 = T_i \qquad \text{for } t = 0 \text{, in } 0 < x < L_1 \tag{1.d}$$

Region 2:

$$C_2 \frac{\partial T_2}{\partial t} = k_2 \frac{\partial^2 T_2}{\partial x^2} \qquad \text{in } L_1 < x < L_2 \text{, for } t > 0 \tag{2.a}$$

$$k_1 \frac{\partial T_1}{\partial x} = k_2 \frac{\partial T_2}{\partial x} \qquad \text{at } x = L_1 \text{, for } t > 0 \tag{2.b}$$

$$k_2 \frac{\partial T_2}{\partial x} = 0 \qquad \text{at } x = L_1 + L_2 \text{, for } t > 0 \tag{2.c}$$

$$T_1 = T_i \qquad \text{for } t = 0 \text{, in } L_1 < x < L_2 \tag{2.d}$$

where $C_i = (\rho c_p)_i$, i=1,2, is the volumetric heat capacity.

The problem defined by Eqs. (1.a-d) and (2.a-d), with known thermophysical properties and known boundary and initial conditions, constitutes a *Direct Heat Conduction Problem*. The objective of the direct problem is to obtain the transient temperature field in regions 1 and 2. The finite-volume method was applied for the solution of the direct problem. The numerical solution obtained here was validated with the analytical solution obtained with the Classical Integral Transform Technique (Özişik, 1993) for representative test-cases.

4. Inverse Problem

For the *Inverse Problem* considered here, the properties C_1, k_1, C_2 and k_2 are regarded as unknown quantities. Note in Eqs. (1.a-d) and (2.a-d) the following independent groups involving these thermophysical properties: (k_1/C_1), k_1, (k_2/C_2) and (k_2/k_1). Therefore, the simultaneous estimation of properties C_1, k_1, C_2 and k_2 appears to be possible. For the estimation of $\mathbf{P}=[C_1, k_1, C_2, k_2]$, we consider available for the inverse analysis the transient readings Y_i taken at times t_i, $i = 1,\ldots,I$, of one temperature sensor located at the heated surface at x=0 (Oliveira *et al.*, 1999, Silva *et al.*, 2000, Salas *et al.*, 2002). In the present paper, the unknown parameters are estimated with the *Levenberg-Marquardt Method* (Beck and Arnold, 1977, Özişik and Orlande, 2000).

For the estimation of the unknown parameters with the Levenberg-Marquardt method, let us assume that the temperature measurement errors are additive, normally distributed with zero mean and known and constant standard-deviation. We also assume that all the other quantities appearing in the formulation of the problem are exactly known for the inverse analysis and that there are no errors in the independent variables. In this case, the least-squares norm becomes a minimum variance estimator (Beck and Arnold, 1977). This norm is written in matrix form as:

$$S(\mathbf{P}) = [\mathbf{Y} - \mathbf{T}(\mathbf{P})]^T [\mathbf{Y} - \mathbf{T}(\mathbf{P})] \tag{3.a}$$

where

$$[\mathbf{Y} - \mathbf{T}(\mathbf{P})]^T = [Y_1 - T_1(\mathbf{P}), Y_2 - T_2(\mathbf{P}), ..., Y_I - T_I(\mathbf{P})] \tag{3.b}$$

The estimated temperatures $T_i(\boldsymbol{P})$ are obtained from the solution of the direct problem given by Eqs. (1.a-d) and (2.a-d) by using estimated values for the unknown parameters. The iterative procedure of the Levenberg-Marquardt Method, for the minimization of the objective function given by Eq. (3.a), is given by

$$\mathbf{P}^{k+1} = \mathbf{P}^k + (\mathbf{J}^T\mathbf{J} + \mu^k\Omega^k)^{-1}\mathbf{J}^T[\mathbf{Y} - \mathbf{T}(\mathbf{P}^k)] \tag{4}$$

where $\mathbf{J}$ is the sensitivity matrix, μ^k is the damping parameter and $\mathbf{\Omega}^k$ is a diagonal matrix, which can be taken as the identity matrix or as the diagonal of $\mathbf{J}^T\mathbf{J}$. The elements of the sensitivity matrix are denoted as the *sensitivity coefficients*. They provide a measure of the sensitivity of the estimated (or measured) temperatures with respect to changes in the unknown parameters. Clearly, the solution of inverse problems involving sensitivity coefficients with small magnitudes is extremely difficult, because very different values for the unknown parameters would result in basically the same value for the measured variables. Also, the columns of the sensitivity matrix are required to be linearly independent in order to have the matrix $\mathbf{J}^T\mathbf{J}$ invertible.

After the minimization of the objective function, given by Eq. (3.a), a statistical analysis can be performed in order to obtain confidence intervals for the estimated parameters. Confidence intervals at the *99%* confidence level are obtained as (Beck and Arnold, 1977, Özişik and Orlande, 2000):

$$\hat{P}_j - 2.576\sigma_{\hat{P}_j} \leq P_j \leq \hat{P}_j + 2.576\sigma_{\hat{P}_j} \qquad j = 1, 2, 3, 4 \tag{5.a}$$

where $\hat{P}$ are the estimated values and $\sigma_{\hat{P}}$ are the standard deviations for the unknown parameters.

The standard deviations are given by

$$\sigma_{\hat{P}_j} = \sqrt{V_{jj}} \tag{5.b}$$

where V_{jj} is the j^{th} element in the diagonal of the covariance matrix for the estimated parameters, $\boldsymbol{V}$. Expressions can be obtained for such matrix in linear estimation problems (Beck and Arnold, 1977). These linear expressions can be approximately used for non-linear cases, such as the one of this work. The covariance matrix of the estimated parameters for the ordinary least-squares norm is given by

$$\mathbf{V} = (\mathbf{J}^T\mathbf{J})^{-1}\sigma^2 \tag{5.c}$$

Optimum experiments can be designed by minimizing the hypervolume of the confidence region of the estimated parameters, in order to ensure minimum variance for the estimates. The minimization of the confidence region can be obtained by maximizing the determinant of $\mathbf{V}^{-1}$, in the so-called *D-optimum design* (Beck and Arnold, 1997, Alifanov, 1994, Özişik and Orlande, 2000, Woodbury, 2002). With the covariance matrix $\mathbf{V}$ given by Eq. (5.c), we can design optimum experiments by maximizing the determinant of the so-called Fisher's Information Matrix, $\mathbf{F} = \mathbf{J}^T\mathbf{J}$.

5. Results and Discussions

We now consider the analysis of the sensitivity coefficients and of the determinant of the information matrix for the estimation of $\mathbf{P}=[C_1, k_1, C_2, k_2]$. For nonlinear estimation problems, such as the one under picture in this work, the analyses of the sensitivity coefficients and of the determinant of $\mathbf{F}$ are not global, because these quantities are functions of the unknown parameters. Therefore, *a priori* estimated values for the parameters are required for the design of optimum experiments. In this case, let us assume C_1=22.73 x 10^5 J/m^3K, k_1=0.35 W/mK, C_2=24.61 x 10^5 J/m^3K and k_2=164 W/mK. For the results presented below, the heat flux at the boundary x=0 is supposed to be q=358 W/m^2, and the heating time is taken as $t_h=t_f$=840 s. In addition, the temperature readings are assumed to be available every 1 *s*. The dimensions of regions 1 and 2 are assumed to be the same as for the experimental apparatus presented above.

Figure 3 presents the transient variations of the normalized sensitivity coefficients with respect to the different parameters, as well as of the temperature, at the measurement location. The normalized sensitivity coefficients are obtained by multiplying the original sensitivity coefficients by the parameters that they are referred to. Figure 3 shows that the normalized sensitivity coefficients with respect to C_1 , k_1 and C_2 attain magnitudes of the same order of the temperature and are not linearly-dependent. Therefore, the conditions are appropriate for the simultaneous estimation of these three parameters. On the other hand, the order of magnitude of the sensitivity coefficient with respect to k_2 is very small. Such is the case because region 2 is made of aluminum and its thermal conductivity is very high. As a result, the temperature in the aluminum cylinder is practically uniform and temperature gradients can be neglected. In other words, a lumped formulation, which is independent of the thermal conductivity, would be appropriate for the mathematical model in region 2.

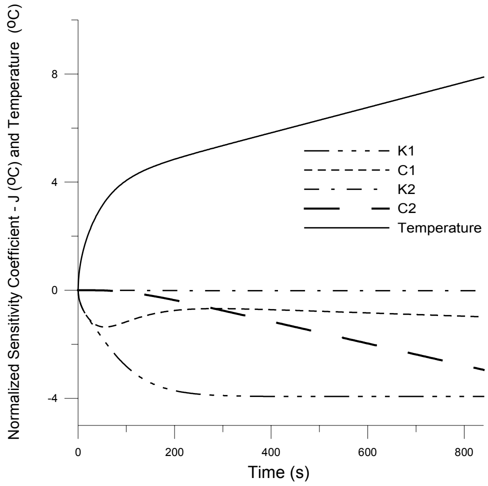

Figure 3. Temperature and sensitivity coefficients

The time variation of the determinant of the information matrix is presented in Fig. 4, for the simultaneous estimation of C_1, k_1, C_2 and k_2. The determinant was computed for the same experimental conditions specified above and for a fixed measurement frequency of 1 reading every 1 s. Figure 4 shows that the determinant undergoes a large increase for t < 400 s, when it is varied by many orders of magnitude. For larger times, the rate of increase of the determinant is gradually reduced. Within the duration considered for the experiment, the determinant maximum value is attained for $t_h=t_f$=840 s.

Before considering the use of actual experimental data for the estimation of the thermophysical properties, let us examine the use of simulated temperature measurements in the inverse analysis of estimating C_1 , k_1 and C_2. In this case, the value of k_2 was set to 164 W/mK. However, we note that uncertainties in the value assumed for k_2 do not affect significantly the estimated parameters, because it sensitivity coefficient is practically null. The simulated measurements contained random errors with standard-deviation of 0.16 ^oC. Table 1 presents the results obtained with the Levenberg-Marquardt method for different initial guesses used for the iterative procedure, by considering the same experimental conditions described above in the analysis of the sensitivity coefficients and the determinant of the information matrix. The parameters were quite accurately recovered, even for initial guesses quite far from their exact values. The estimated parameters were not affected by the initial guesses in the range examined. We note that the parameters were exactly recovered when errorless measurements were used in the inverse analysis.

We now examine the use of actual measured data in the inverse analysis. As for the case examined with simulated measurements, the value of k_2 was set to 164 W/mK for the solution of the inverse problem. The transient measurements of the heat flux and of the temperature at the heated surface (x=0) were used for the estimation of C_1, k_1 and C_2. In the experiments, the data acquisition system was first started in order to record the initial temperature in the region. After approximately 60 seconds, the heating was started by turning on the DC power source, with a previously set voltage of 30 V. The instant when the heating was started could be determined through the readings of the heat flux transducer. Figures 5.a presents the temperature readings of the thermocouples located at $x=0$ *mm*, x=L_1=4.4 *mm* and x=L_1+L_2 = 31.4 *mm*. Note in this figure that the readings of the thermocouples located at L_1 and at L_1+L_2 are practically identical, because of the aluminum high thermal conductivity. Figure 5.b presents the measured heat flux for the same experimental run of Fig. 5.a. Note in Fig. 5.b that the heat flux does not become steady during the experiment, because of the finite volumetric heat capacities of the electrical resistance and of the transducer.

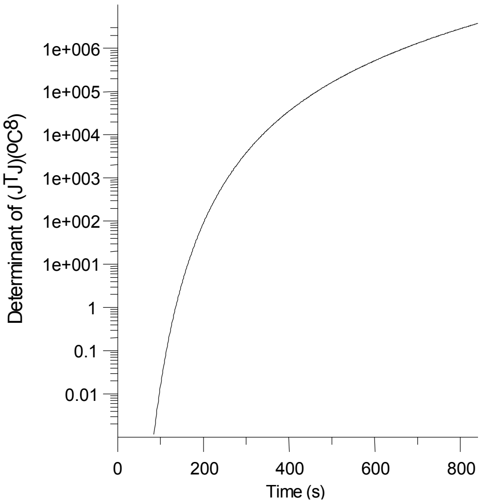

Figure 4. Determinant of the information matrix

Table 1. Parameters estimated with simulated measurements

Estimation	Parameter	Initial Guess	Estimated Parameter	Standard Deviation
1	K_1 *(W/mK)*	0.30	0.35	1.95×10^{-3}
	C_1 *(J/m³K)*	24.61×10^{5}	22.73×10^{5}	0.43×10^{5}
	C_2 *(J/m³K)*	22.73×10^{5}	24.61×10^{5}	0.17×10^{5}
2	K_1 *(W/mK)*	0.10	0.35	1.95×10^{-3}
	C_1 *(J/m³K)*	34.61×10^{5}	22.73×10^{5}	0.76×10^{5}
	C_2 *(J/m³K)*	12.73×10^{5}	24.61×10^{5}	0.27×10^{5}
3	K_1 *(W/mK)*	1.00	0.35	5.41×10^{-3}
	C_1 *(J/m³K)*	34.61×10^{5}	22.73×10^{5}	0.70×10^{5}
	C_2 *(J/m³K)*	32.73×10^{5}	24.61×10^{5}	0.20×10^{5}

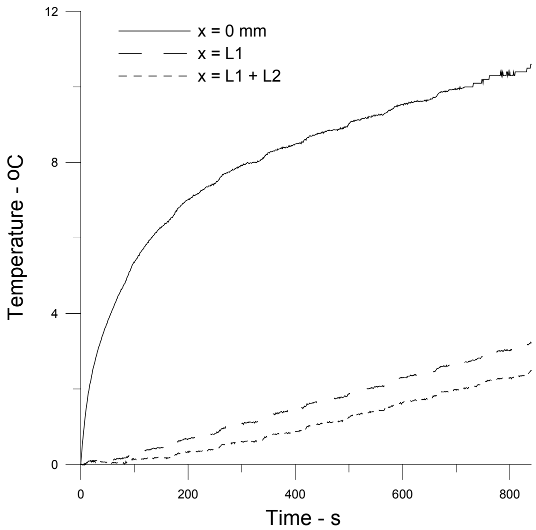

Figure 5.a – Temperature measurements

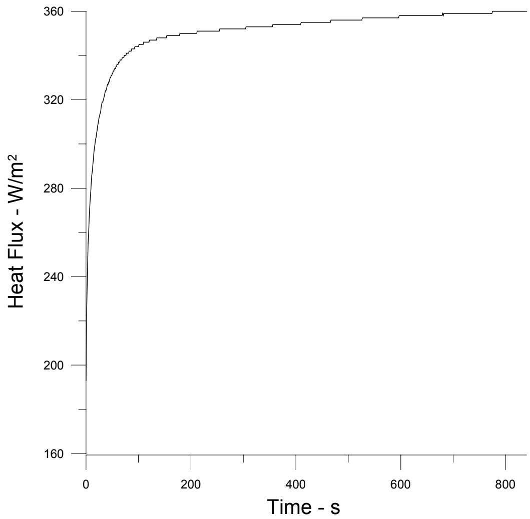

Figure 5.b – Heat flux measurements

By using the measurements of the temperature at x=0 *mm* and of the imposed heat flux, which are shown in Figs. 5.a,b, the values presented in Table 2 were estimated for the unknown properties. Table 2 shows the results obtained with different initial guesses used for the Levenberg-Marquardt method. We note that the estimated values are not affected by the initial-guesses examined. In addition, the repetition of the experiment resulted on the same values estimated for the parameters.

The residuals between the measured and the estimated temperatures, obtained with the values estimated for the parameters shown in table 2, are presented in Fig. 6, for the sensors located at x=0 *mm*, $x=L_1$=4.4 *mm* and $x=L_1+L_2$ = 31.4 *mm*. We note that only the measurements of the sensor at x=0 *mm* were used in the inverse analysis, but the residuals for all sensors are presented here in order to assess the accuracy of the estimated parameters. Figure 6 shows that the residuals for the sensor at x=0 m are quite small and practically non-correlated. Such result reveals a significant improvement over our previous results (Salas *et al.*, 2002), due to the more appropriate mathematical model for the physical problem and due to the more controlled experimental conditions. The residuals for the other sensor locations are also small, but they tend to be correlated, specially at x=L_1+ L_2 = 31.4 *mm*. The correlation for the residuals at such position is due to heat losses at the non-heated boundary, which was assumed as thermally insulated in the mathematical model (see equation (2.c)). However, we note that the sensitivity of the estimated parameters with respect to heat losses at this surface was very small.

Table 2 – Parameters estimated with actual measurements

Estimation	Parameter	Initial Guess	Estimated Parameter	Standard Deviation
1	K_1 *(W/mK)*	0.30	0.200	0.80 x 10^{-3}
	C_1 *(J/m³K)*	24.61 x 10^5	23.17 x 10^5	0.26 x 10^5
	C_2 *(J/m³K)*	22.73 x 10^5	26.47 x 10^5	0.21 x 10^5
2	K_1 *(W/mK)*	0.10	0.200	0.80 x 10^{-3}
	C_1 *(J/m³K)*	34.61 x 10^5	23.17 x 10^5	0.24 x 10^5
	C_2 *kJ/m³K)*	12.73 x 10^5	26.47 x 10^5	0.32 x 10^5
3	K_1 *(W/mK)*	1.00	0.200	0.80 x 10^{-3}
	C_1 *(J/m³K)*	34.61 x 10^5	23.17 x 10^5	0.24 x 10^5
	C_2 *(J/m³K)*	32.73 x 10^5	26.47 x 10^5	0.33 x 10^5

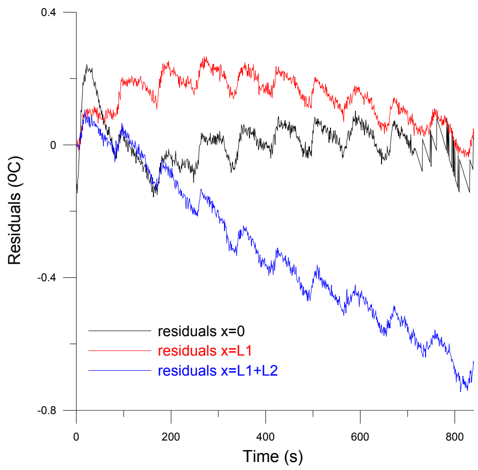

Figure 6. Residuals of estimation 3

Finally, the thermophysical properties estimated above are compared to the results obtained with other techniques. The thermal conductivity and volumetric heat capacity for Teflon, identified with the Flash method (Massard *et al.*, 2004), at the temperature range of 25 to 75 ^oC, are presented in Table 4. The thermal conductivity identified with the same experimental apparatus described in this work, but by using the steady-state heat flux and temperature difference across the Teflon cylinder, was 0.22 ± 0.01 *W/mK*, at the average temperature of 60.14 ºC (Soares, 2005). Therefore, the thermal properties identified with the different techniques, in the Laboratory of Heat Transmission and Tehcnology, COPPE/UFRJ, are in excellent agreement.

Table 4 – Teflon properties measured with the Flash method

Temperature (^oC)	Conductivity (*W/mK*)	Thermal Capacity (*J/m³K*)
25	0.19 ± 0.01	21.71 x 10^5
50	0.235 ± 0.003	(23.51 ± 0.01) x 10^5
75	0.302 ± 0.005	(30.51 ± 0.05) x 10^5

6. Conclusions

In this paper, we presented the identification of thermophysical properties of solids, by using inverse analysis techniques and transient measurements of temperature and heat flux. The Levenberg-Marquardt method was used for the solution of the present parameter estimation problem and the experiment was designed by using the D-Optimum approach. The experimental apparatus and the mathematical model were advanced from previous works performed in our laboratory. The results obtained with the technique developed were not sensitive to the initial guesses used for the Levenberg-Marquardt method and were not affected by repetitions of the experiment. Furthermore, they are in excellent agreement with those results obtained with other experimental techniques, including the Flash method.

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9. Responsibility notice

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